apparatus actually constructed, the whole moving system is enclosed in an evacuated chamber in order for it to be free from disturbances due to the buoyancy change of the sample, the air flow around the balance, and the condensation of water vapor on the suspension system at low temperatures.

For example, Figs. 3 and 4 show a recorded chart of the compensating current in the case of nickel and the calibration curves

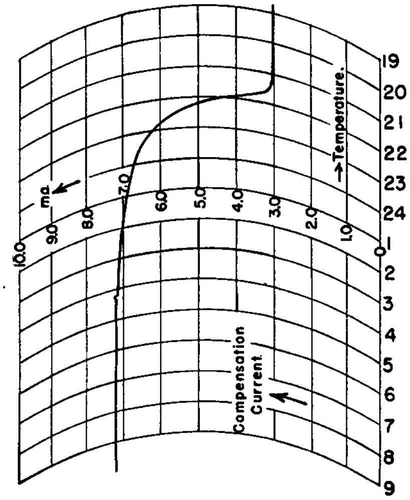

FIG. 3. Recorded chart of the change of saturation magnetization of Ni due to the temperature change.

for the relation between the compensation currents and weights.

Improved characteristics of the present magnetic balance are as follows.

(1) The range of operating temperature can be easily extended The operation for the temperature change of the sample in the range from liquid He to about 1000°C is very easily performed by using a small furnace or Dewar vessel.

(2) Exchange of samples needs only the readjustment of the counter weight according to the mass difference of the samples, and no other adjustment is required.

(3) The adjustment and the calibration for the measurement are simpler than those of the previously reported ones. The stability of the whole apparatus is so good that the readjustment of the main part is not required even after several days. Therefore,

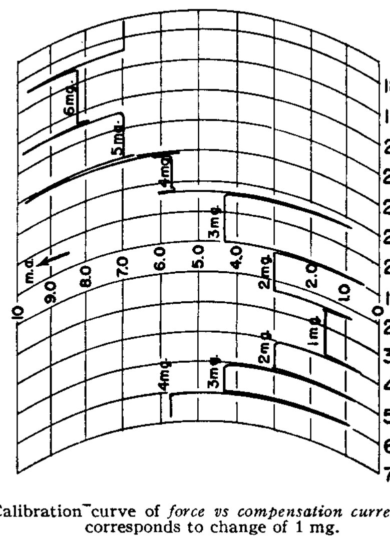

FIG. 4. Calibration curve of *force vs compensation current*, each stage corresponds to change of 1 mg.

the calibration is not required for each measurement except in the case of especially accurate measurement.

(4) The mechanical disturbance of short period, such as the effect of the operation of the rotary pump or rough walking in the neighborhood of the apparatus makes no influence on the measurement.

(5) The sensitivity of this balance can be brought to sufficiently higher order for wide range of the mass of sample. In ordinary cases the sensitivity is of the order of 1 mg per ma, and it is adjustable by changing the order of feedback efficiency.

* This research was supported partly by a Grant-in-Aid for Fundamental Scientific Research by the Ministry of Education.

[1] Eisler, Newton, and Adcock, Rev. Sci. Instr. **23**, 17 (1952); R. F. S. Ropertson and P. W. Selwood, Rev. Sci. Instr. **22**, 146 (1951); H. A. Allred, Rev. Sci. Instr. **19**, 818 (1948); J. W. Clerk, Rev. Sci. Instr. **18**, 915 ff (1947).

[2] Curie, Ann. chim. et phys. **5**, 289 (1895).

Techniques Useful in Evacuating and Pressurizing Metal Chambers*

H. V. NEHER AND ALAN R. JOHNSTON
California Institute of Technology, Pasadena, California
(Received February 22, 1954)

WHEN metal chambers are to be evacuated and baked out and then either left evacuated or filled with a gas, the following problems are apt to arise: (1) If the container is to be sealed off, how shall it be sealed, especially with pressure inside, and (2) if equipment is to be mounted inside how shall the opening be sealed? The procedures to be described are not new but are apparently not generally known. They are so simple and useful that it is thought desirable to call the experimentalist's attention to them.

The aforementioned problems arose in connection with the automatic ionization chamber described previously.[1] It was found that the functioning of these instruments was more consistent if the ion chamber was baked at 350°C for several hours and then filled with pure argon. This procedure resulted in the elimination of organic impurities from the inner surfaces. The impurities, settling on the gold-coated quartz fiber and the collector, apparently altered the contact, resulting in some uncertainty in the recharging. The baking procedure has completely curved this difficulty.

In making the ionization chamber, commercial radio-tube parts were used.[1,2] The convolute on the lip of the copper-plated steel shell has a matching silver-plated convolute in the metal part of the header as shown in Fig. 1. The header has 8 conductors passing through its glass button giving an easy means of making connecnections to the equipment mounted inside. The seal between the two parts is made by using a 0.005 in. thick copper gasket clamped between the two convolutes and the whole pressed tightly together with two steel clamping rings. The rings shown in Fig. 1 are $\frac{5}{32}$ in. thick, $\frac{1}{4}$ in. wide, and contain 12 equally spaced screws.

It is quite simple to secure a leakproof seal by this method. In the case of either copper-to-copper or silver-to-copper a diffusion weld occurs during the baking process and considerable difficulty is sometimes experienced, when it is desired to break the seal, in removing the gasket where it comes in contact with the convolute. It is, of course, not necessary to use commercial radio-tube parts for the seals. Turned copper or copper-plated steel parts, with convolutes properly placed to give high pressure on the copper gasket, will do equally well.

Such a seal is good for either vacuum or pressure. The temperature at which the apparatus may be baked is limited by the glass button in the header which will withstand about 375°C. The maximum pressure a header of this type will safely withstand, when cold, is about 300 lb in.$^{-2}$. Pressures of 120 lb in.$^{-2}$ are regularly used in the ion chambers.

The other problem, of sealing the tubulation through which evacuation and filling take place, is solved by using a copper tube

Downloaded 27 Jul 2006 to 131.215.225.171. Redistribution subject to AIP license or copyright, see http://rsi.aip.org/rsi/copyright.jsp

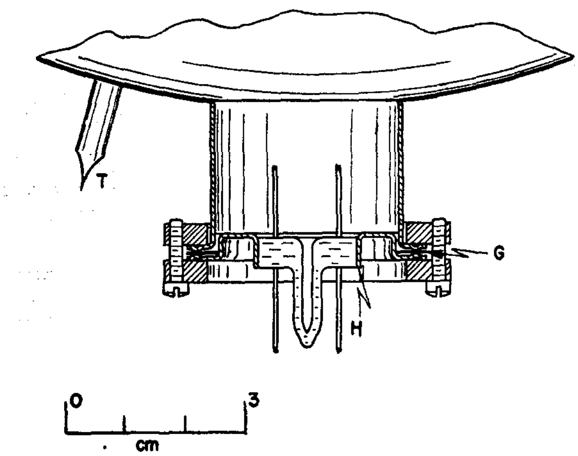

FIG. 1. The copper gasket G makes a vacuum- or pressure-tight seal between the plated steel convolutes when pressed together by means of the clamping rings. The header H is a commercial radio-tube part (6L6) that has 8 conductors passing through the glass button. The copper tubulation T is sealed against pressure or vacuum by the pinch-off tool shown in Fig. 2.

and a pinch-off tool, forming a cold weld. Such tools are in fairly common use by the vacuum tube industry but seem not to be commonly known in many research laboratories. The one here described was made in our shops and will satisfactorily seal off copper tubing of up to $\frac{5}{16}$-in. outside diameter with either a vacuum inside or gas pressures up to several hundred pounds per sq. in.

To make a cold weld satisfactory at such a seal very high pressures must be used. The clean, soft copper will, under these circumstances, adhere to itself, forming a diffusion weld.

The tool is designed to give a high mechanical advantage during the last part of the travel of the jaws. These jaws, in the tool here described, are $\frac{1}{2}$-in. in diameter, are made of drill rod, and are extremely hard. They are so mounted that when they make contact they are accurately parallel. The arm M, Fig. 2, has mounted in its top side, opposite the movable jaw, a hardened steel insert C, whose face has a radius of curvature only slightly greater than the distance AC. The hardened roller mounted in the lever L moves along the flat portion of this insert at the beginning of the pinch-off operation, and then along the curved part as the jaws close. It is obvious that if the radius of curvature of C and its center of curvature were AC and at A, respectively, no motion of the jaw would take place as the roller moved along the curved portion of C. Control over the position of the center of curvature is made possible by means of the adjusting screw.

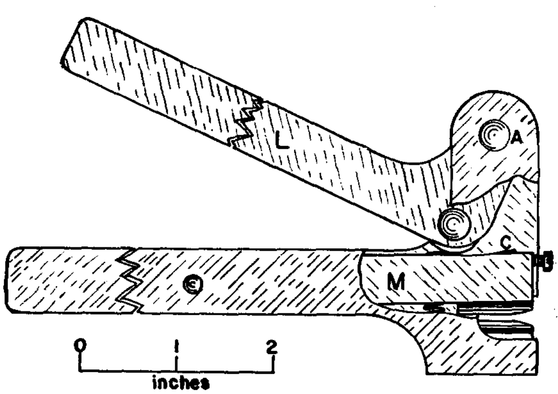

FIG. 2. The pinch-off tool is designed to give a large mechanical advantage when the jaws are nearly closed. Such a tool will pinch off copper tubing of up to $\frac{5}{16}$-in. o.d., giving a diffusion weld.

A further technique, useful where copper parts can be used, is that of making brazed joints by means of an alloy of copper and phosphorus. This material, made under the trade name of "Phos-copper,"[3] melts at 750°C and contains sufficient phosphorus to act as a reducing agent when used on copper. Brazed joints thus made are free from the usual contamination of fluxes. This alloy is useful in connecting the piece to be evacuated to the vacuum manifold as well as in the fabrication of equipment generally.

* Assisted by the joint program of the U. S. Office of Naval Research and the U. S. Atomic Energy Commission.

[1] H. V. Neher, Rev. Sci. Instr. 24, 99 (1953).
[2] Type 6L6 parts obtainable from Radio Corporation of America, Harrison, New Jersey.
[3] Made by Westinghouse Manufacturing Company, Pittsburgh, Pennsylvania.

A Cryostat for Adsorption Experiments

J. A. MORRISON AND D. M. YOUNG*
Division of Pure Chemistry, National Research Laboratories, Ottawa, Canada
(Received February 8, 1954)

IN the measurement of adsorption isotherms of gases and vapors on solids below room temperature, it is customary to use thermostatted liquid baths or baths of freezing mixtures. Most low-temperature measurements are made with baths of liquid nitrogen, oxygen, or air, and control of these over a narrow temperature range to ±0.01°K has been achieved[1,2] without too much difficulty. However, for experiments over the large temperature range from 90°K to room temperature no suitable single thermostat has been devised. The experiments can be done in a low-temperature calorimeter, modified for adsorption work,[3,4] but such an apparatus is best suited for exhaustive measurements with a single solid substrate. Jura and Criddle[5] have used a cryostat after the design of a low-temperature calorimeter for adsorption measurements between 63° and 77°K.

The purpose of this note is to point out that temperature control to 0.001° for a period of hours at any temperature between 60° and 300°K can be readily attained with a relatively simple cryostat. The principle of the cryostat is to attach the adsorption cell to a block of metal of large heat capacity which is well insulated from its surroundings. In the model to be described, the block is hung within an evacuated can (pressure -1×10^{-5} mm Hg or less) immersed in liquid air in a Dewar vessel. Loss of energy from the block by radiation and conduction to its surroundings is compensated for by manual control of the current passed through a heater wound on the block. Since the thermal inertia of the block is large, automatic control of the heater current is hardly necessary. There are no critical dimensions in the design, hence the physical arrangement may be varied within wide limits.

The cutaway drawing, Fig. 1, illustrates a form of the cryostat which we have found convenient to use. Constructional details are given in the diagram but a few comments on the operation may be made.

(1) The block is cooled by admitting a small pressure of exchange gas (helium or hydrogen) to the vacuum space. To cool from room temperature to 90°K requires 4 to 5 hours. Temperatures below 90°K are attained by pumping on the liquid air. (If temperatures below about 90°K are not desired, the outer case assembly can be dispensed with.)

(2) Control of the current through the heater (100 ohms of No. 30 D.G.C. constantan) is achieved by means of a potential divider across a Variac. Using the ordinary laboratory power supply, it was found necessary to adjust the current slightly as often as every 10 or 15 minutes in order to maintain the temperature within 0.001°. With a controlled voltage supply virtually no adjustment was required. On several occasions the system was left overnight on controlled voltage and the total drift in temperature in this time never exceeded 0.02°. This also shows the insensitivity of the temperature control to the level of the liquid air in the Dewar vessel.

Downloaded 27 Jul 2006 to 131.215.225.171. Redistribution subject to AIP license or copyright, see http://rsi.aip.org/rsi/copyright.jsp